Micro-XRF Analysis of Inclusions in Plastic Films

Introduction

Plastic films are widely used for a large variety of industrial products. Their required quality must be very high for certain applications. Uniformity of such parameters as thickness and composition can be crucial. The presence of inclusions can be seriously detrimental to performance characteristics of the desired film.

The origin of inclusions is typically material from either the manufacturing equipment or other contamination. Micro X-ray Fluorescence (Micro-XRF) analysis of an inclusion can provide the following information:

- Element or alloy composition and thereby often the source of the inclusion. With this information, optimization of the manufacturing process may be possible.
- Condition of the manufacturing equipment. For example, the identified inclusion could be traced to a failing component.
- Quality of source materials and/or process problems.

The Analytical Problem

Very often the ability to analyze microscopically small inclusions rather than a bulk volume enables definitive identification of what would otherwise be diluted to the point of non-detection. The inclusion represents only a very small concentration over the complete area of the film. Other methods of analysis involve the dissolution of the film, which can be very time consuming.

An effective analytical method is one that works non-destructively, provides information for a wide range of elements, and has a lateral resolution that can focus on single inclusions. Energy Dispersive X-ray Spectroscopy (EDS) methods satisfy these requirements, especially when coupled with small area excitation techniques.

Measuring Instrument

The Orbis Micro-XRF Elemental Analyzer provides small area X-ray excitation by a total reflectance capillary optics system, situated between the X-ray source and the sample. Because the entrance of the capillary is very close to the X-ray tube, a wider angle of radiation can be captured, compared to standard collimator methods. This results in only small losses of intensity on the sample.

The characteristic fluorescent X-ray radiation from the sample is measured with an energy dispersive Silicon Drift Detector (SDD) and associated spectral processing electronics. SDDs provide high throughput and high resolution spectral data but do not require liquid nitrogen for cooling.

The analyzed area can also be observed by a video microscope and positioned by a computer controlled X-Y-Z stage.

The Orbis Micro-XRF Elemental Analyzer

Measuring Conditions

Excitation Voltage	40 kV
Current	0.5mA
Tube Anode	Mo
Measuring Time	300 s
Spot Size	300 µm
Positioning Accuracy	10 µm
Detector	30 mm^2 SDD
Resolution	≤ 135 eV, Mn-Kα
Optical Path	Vacuum

Sample Preparation

The films were supported over the end of plastic sample cells and directly measured. The areas of inclusions were positioned to prevent the possibility of scattered radiation from the cell wall interfering with the measurements.

Measurements were made on pure films, as well as the inclusions. Correct positioning of the sample was simplified using the video microscope.

Results

Figure 1 compares spectra obtained on a pure film (red filled), with that from an area containing an inclusion (black outline). Clearly, the inclusion contains the elements Fe, Mn, and Cr. The intensity relationship for these peaks suggests that this inclusion is steel based (probably a stainless steel alloy).

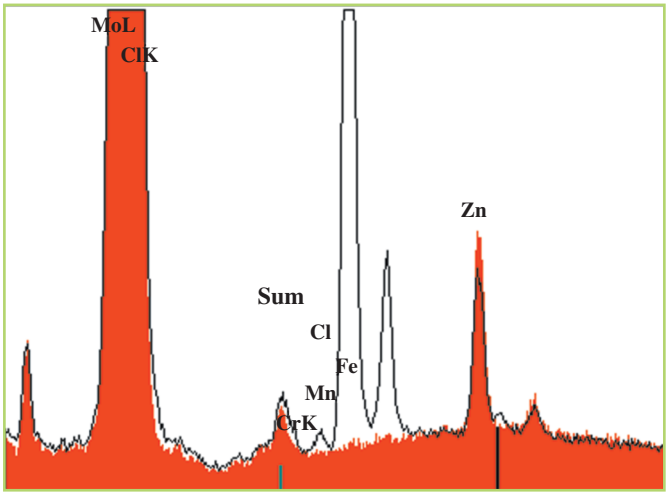

Figure 1. Comparison of spectra from inclusion and film.

In fact, in this example, the inclusion was indeed a steel particle with a diameter of approximately 100 µm. In Figure 2, a similar spectral comparison is shown for an inclusion that was considerably smaller. In fact, no actual particle could be seen, even with a high magnification (100x). The inclusion was only apparent as a visual non-homogeneity of the film.

The differences in comparative intensities are consequently much smaller, but it can still be seen that for elements to be expected in steel (in particular the Fe), intensities from the "inclusion" are higher than those for the blank sample.

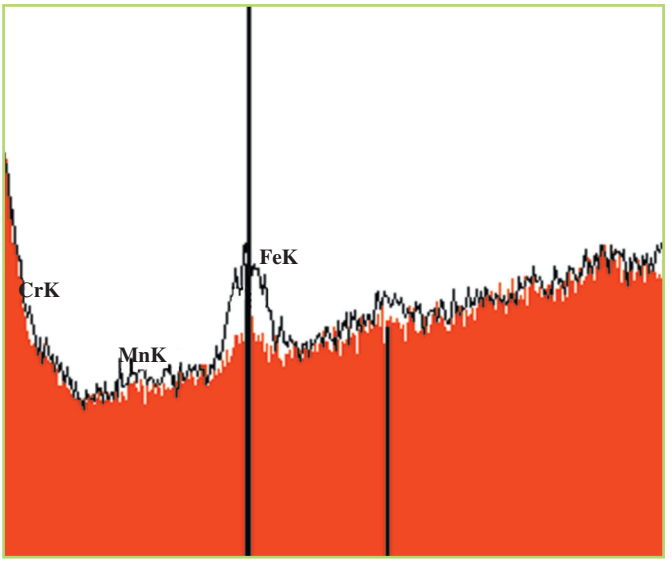

Figure 2. Comparison of spectra from (<10 µm) inclusion and film.

Conclusion

By limiting the incident X-ray beam within a small area, analysis with lateral resolution is possible. The high intensity of this focused incident beam makes it possible for high sensitivities to be achieved for small analysis areas.

The analytical problem discussed here shows that inclusions in plastic films can be identified, provided their elemental composition is within the element range covered by standard EDXRF methods.

Also, in the case where no particles are actually visible, the composition of the observed non-homogeneity in the film still provides elemental identification and strong evidence as to a likely source of a defect.